Indian J. Phys. **52A**, 578–580 (1978)

A preliminary report on the crystal structure of α—α′-dipyridil iminodiacetato oxovanadium (IV)

MINAKSHI GHOSH

Department of Magnetism,
Indian Association for the Cultivation of Science, Calcutta-700032

(*Received* 21 *March* 1978)

In an attempt to obtain planned heterochelates of oxo-vanadium (IV), several dibasic tridentate ligands, along with bidentate neutral donors, could be used with success (Dutta & Ghosh 1967). One of the compounds prepared in this project is α-α′-dipyridil-iminodiacetato-oxo-vanadium (IV), $VO(C_4H_3NO_4)(C_{10}H_8N_2)$. The crystals of this compound were taken up for a complete X-ray analysis, to find out (i) the nature of co-ordination of V, and (ii) whether the structure consists of polymeric chains or individual complexes. Some preliminary results of this structural study are presented here.

Well formed, shining deep brown needle shaped crystals of the compound were mounted along the elongated axis. While there was no 'horizontal' mirror in the oscillation photograph, the zero-layer Weissenberg photograph displayed two mirror lines, 90° apart. Further, oscillation photograph about another direction perpendicular to the needle axis showed a horizontal mirror, pointing to the conclusion that the crystal belongs to the monoclinic system, the needle axis being one of the nonunique axes while the other one is the *b*-axis of the crystal.

Following cell-dimensions were obtained from the oscillation and Weissenberg photographs

$$a = 11{\cdot}50 \pm {\cdot}03\text{Å}$$
$$b = 15{\cdot}27 \pm {\cdot}01\text{Å} \qquad \beta = 101{\cdot}1 \pm 4^\circ$$
$$c = 8{\cdot}81 \pm {\cdot}04\text{Å}$$

Density of the crystal was determined by flotation method using a mixture of benzene (density = 0·88 gm cm^{-3}) bromoform (density = 2·89 gm.cm^{-3}) and was found to be 1·54 gm.cm^{-3} while that calculated on the basis of $Z = 4$ was 1·55 gm cm^{-3}.

Examination of Weissenberg photographs revealed that $0k0$ (k = odd) and $h0l$ (h = odd) reflections were systematically absent. Thus the space group of the crystal was unequivocally established as $P2_1/a$.

The structure analysis of this crystal evidently calls for the usual heavy-atom method, but some difficulty is apprehended in this procedure due to the

fact that the atomic number of vanadium is only 23 against a total of 168 for one formula unit. Thus too many of the signs calculated on the basis of the vanadium position may be incorrect. Some extra information regarding the structure supplementing those available from the Patterson map would therefore be of help. One such information could be obtained easily, as described below.

The dipyridil molecule consists of two planar pyridine rings linked up by a *C-C* bond and tilted with respect to one another about an axis along the said bond. Now, the dimensions of the unit cell suggest that at least one of the two rings should be almost parallel to the *ab* plane. The *hk*0 section of the weighted reciprocal lattice would thus show up the transform of this ring.

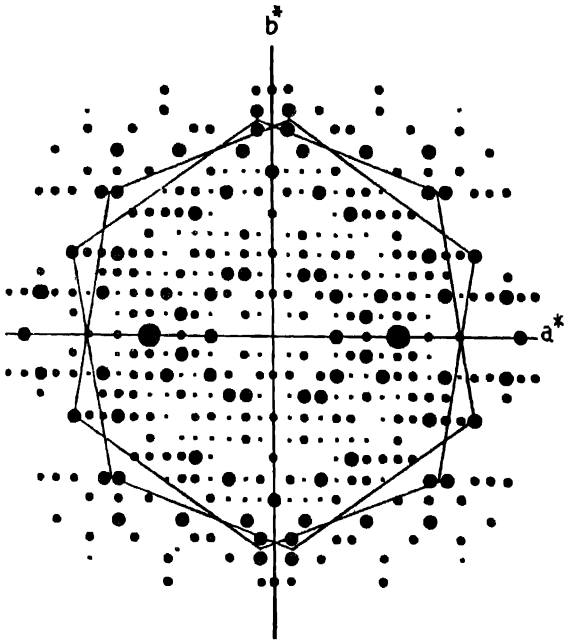

Fig. 1a

Fig. 1(a) shows the *hk*0 section of the reciprocal lattice, weighted by the appropriate unitary structure factor observed at each point. Transforms of six-membered rings in two orientations connected by reflection symmetry are

outlined In Fig 1(b), projection of one of them, as deduced from the transform, is shown Thus the orientation of one of the two rings of the dipyridil molecule could be found out.

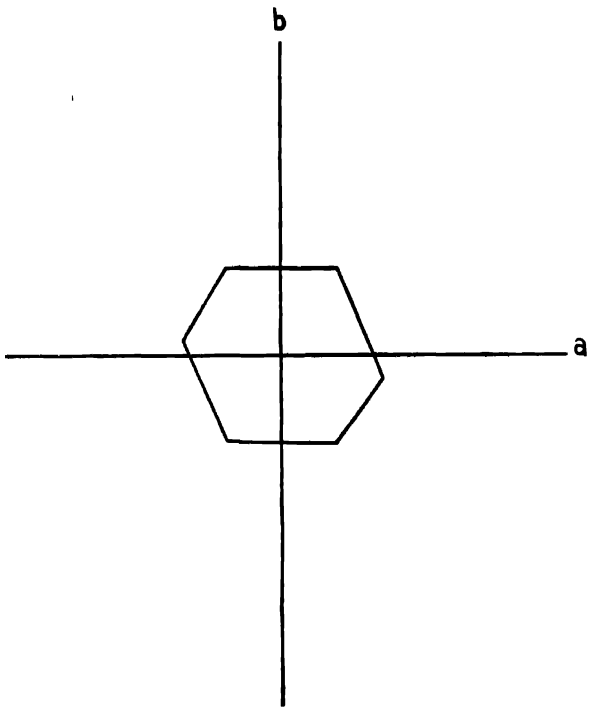

Fig 1b

The author is grateful to Dr Saktiprasad Ghosh of the Department of Inorganic Chemistry for supplying the crystals, and to Dr. Siddhartha Ray for guiding this investigation.

References

Dutta R L. & Ghosh S. 1967 *Jour. Indian Chem. Soc* **44** 306